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Results From the Physics of Colloids Experiment on ISS

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This report is a formal draft or working paper, intended to solicit comments and ideas from a technical peer group.

This report contains preliminary findings, subject to revision as analysis proceeds.

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Abstract

The Physics of Colloids in Space (PCS) experiment was accommodated within International Space Station (ISS) EXpedite the PRocessing of Experiments to Space Station (EXPRESS) Rack 2 and was remotely operated from early June 2001 until February 2002 from NASA Glenn Research Center's Telescience Support Center (TSC) in Cleveland, Ohio, and from the remote site at Harvard University in Cambridge, Massachusetts. PCS was launched on 4/19/2001 on Space Shuttle STS-100. The experiment was activated on 5/31/2001. The entire experimental setup performed remarkably well, and accomplished 2400 hours of science operations on-orbit.

The sophisticated instrumentation in PCS is capable of dynamic and static light scattering from 11 to

169 degrees, Bragg scattering over the range from 10 to 60 degrees, dynamic and static light scattering at low angles from 0.3 to 6.0 degrees, and color imaging.

The long duration microgravity environment on the ISS facilitated extended studies on the growth and coarsening characteristics of binary crystals. The de-mixing of the colloid-polymer critical-point sample was also studied as it phase-separated into two phases. Further, aging studies on a col-pol gel, gelation rate studies in extremely low concentration fractal gels over several days, and studies on a glass sample, all provided valuable information. Several exciting and unique aspects of these results are discussed here.

Introduction

Colloids can be defined as fluids with other particles dispersed in them, particularly particles of size between one nanometer and one micrometer. At the lower bound, the particle sizes are on the order of molecular dimensions, and at the upper bound external forces such as gravity are more important than Brownian motion. The variations in sizes, shapes (spheres, rods, etc.), the volume fractions of the particles involved, the surface charge types and distributions, and the properties of the fluid medium, lead to diverse colloidal systems with several technical applications. It is fascinating to see the resulting widespread colloids phenomenon in nature and in industrial processes. Aerosols, foam, paints, pigments, cosmetics, milk, salad dressings, and several electro- and magneto-rheological fluids are examples of colloidal dispersions or suspensions. Abundant biomedical applications for colloidal crystals are being developed; some examples are drug delivery, biomimetic assemblies, cell encapsulation, tissue culture, and controlled release of drugs, flavors, nutrients, and fragrances. Optical, and information and computer technologies can benefit from "colloid engineering" of novel materials such as photonic crystals, via self-assembly in microgravity.

Starting with well-characterized particles, we can fine-tune the interactions among them to vary from highly repulsive, to weakly attractive, to strongly attractive interactions. Understanding the various mechanisms involved in these interactions is an important science objective in the very wide field of colloids research. In addition, the colloidal particles can serve as model systems for the study of fluid and solid properties since they can be considered to be analogous to atoms; hence, they are of interest in the study of the nature of and transitions among gaseous, liquid, solid/crystal, and amorphous states of matter.

In this PCS experiment, four classes of colloidal samples, namely, binary colloidal crystals, col-pol mixtures, fractal gels, and a glass sample were investigated. Quantitative data on nucleation and growth in the absence of gravity were obtained.

Hardware and Diagnostics

The PCS experiment hardware is comprised of an Avionics Section unit and a Test section unit. They are accommodated side by side in ISS EXPRESS Rack 2, occupying four Middeck Locker Equivalents of rack volume (Figure 1). The PCS hardware uses the EXPRESS Rack utilities of power, air-cooling, water-cooling, and communication for commanding and data telemetry. The PCS Avionics Section provides power

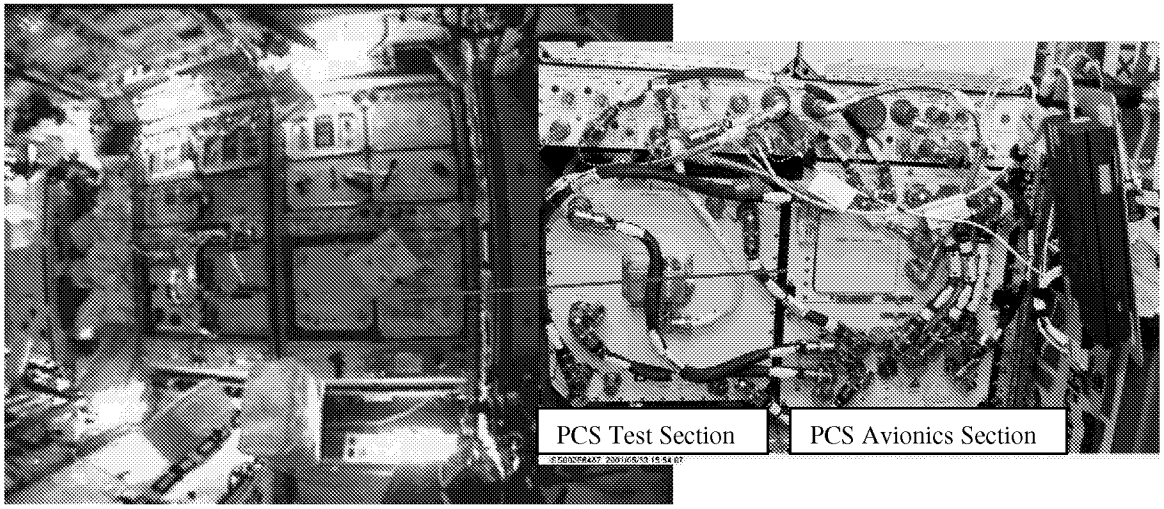


Figure 1 PCS on ISS

distribution, command and data communication, data acquisition and processing, and data storage on 18 GB removable hard drives.

The PCS Test Section contains eight colloid samples and all the diagnostic instrumentation. A schematic of the PCS science diagnostics is shown (Figure 2). These diagnostics are mostly light scattering instrumentation, which were developed substantially under a previous flight experiment, the Physics of Hard Spheres Experiment (PHaSE).[1,2] Dynamic and Static Light Scattering (aka fiber scattering) is provided via a 532 nanometer Nd-YAG laser and fiber-coupled single photon counting detectors. Two detection fibers simultaneously collect light at scattering angles from 11 to 169 degrees and the complementary angle. Bragg scattering is measured over the range from 10 to 60 degrees by imaging the scattering from a second Nd-YAG laser on an optical screen with a digital camera. Additional optics and a second digital camera capture the laser light scattered at low angles of 0.3 to 6.0 degrees. Via the electronics and data processing provided by the Avionics Section, both static and dynamic light scattering data are obtained from the low scattering angle optics and camera. Static light scattering refers to the measurement of the average angular distribution of the scattered light. This distribution is a measure of the Fourier transform of the mass-to-mass correlations in the sample, meaning that this type of measurement provides information about the sizes or positions of the colloids or structures formed and how they are arranged on length scales up to approximately 5 microns. Dynamic light scattering is a technique that measures the spectral width or time dependence of the scattered light, resulting from the motions of particles or structures. Two color cameras (1× and 10× magnification) provide real-space images on macroscopic length scales (to complement the Fourier-space light scattering on microscopic length scales). A further detailed description of the PCS hardware has been given by Ansari et al.,[3]; the detailed description of the PCS experiment objectives has been discussed by Doherty et al.,[4] and a detailed description of the flight hardware operations on ISS is discussed by Doherty, et al.[5]

Thus, the apparatus is equipped to perform seven different measurements[6] – Static Light Scattering (SLS), Dynamic Light Scattering (DLS), High Angle Scattering or Bragg Scattering (HAS), Low Angle Static Scattering (LAS), Low Angle Dynamic Scattering (LAD), Rheology, and Imaging.

Sample Fluids

Table 1 shows the details of the samples used in PCS. A total of eight colloid samples, each approximately 3 milliliter in volume, were used. Each sample was contained in a glass cell that was stationed within a remotely controlled carousel inside the PCS Test Section.

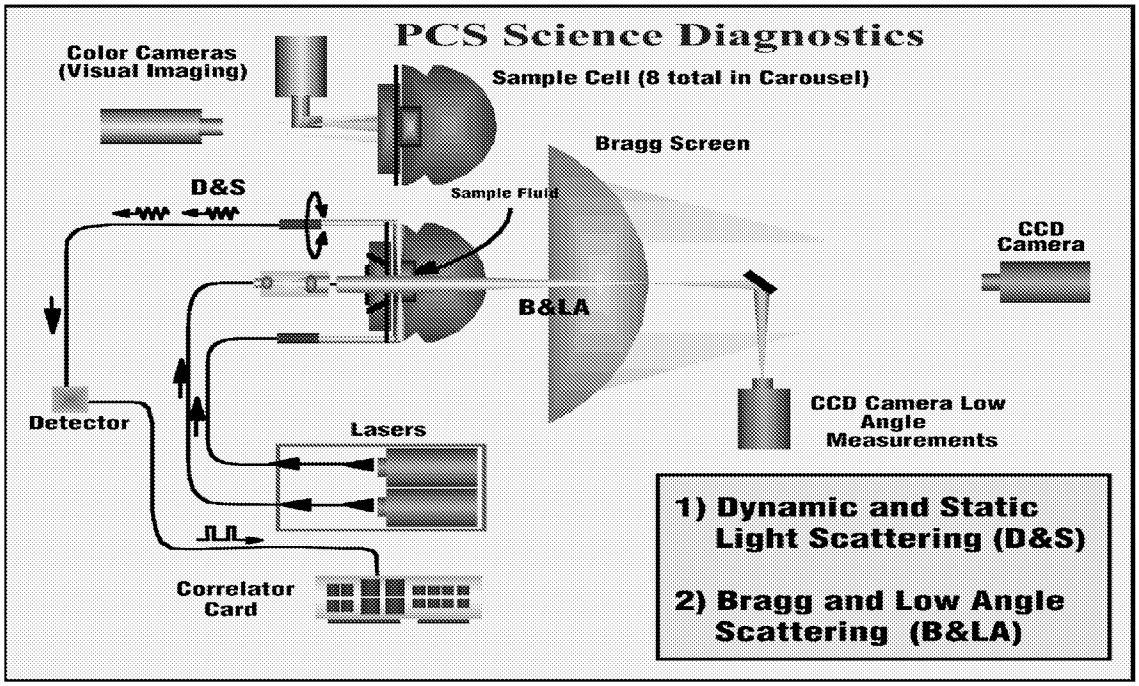


Figure 2 Schematic of the PCS Diagnostics

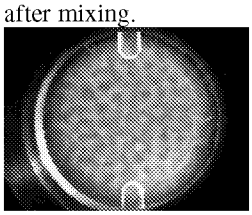
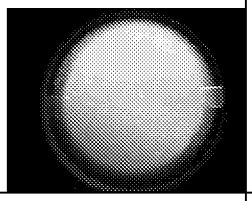
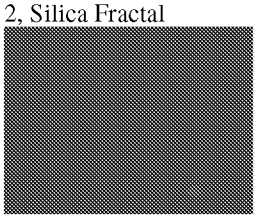
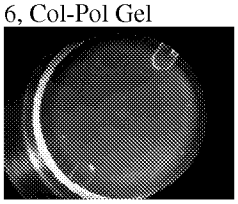
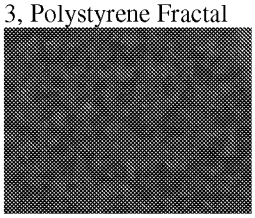
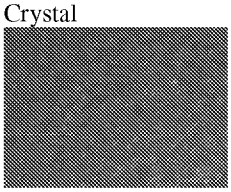
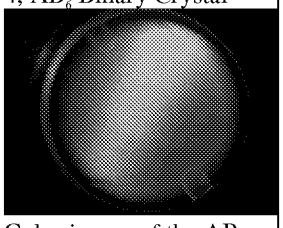
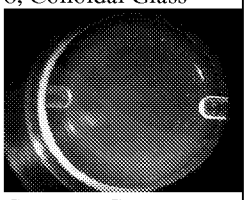
Cell # , Sample Name, sample image	**Sample Details**	**Cell # , Sample Name, sample image**	**Sample Details**
1, Col-Pol Critical Point sample: 11hrs:45mins after mixing. 	r=170 nanometer Colloid and Polystyrene Polymer (Mw=13.2e6); Volume fraction is Colloid: 0.22 Polymer concentration is Cp =1.285 mg/cm3 Solvent is cis-decalin and tetralin – index match particles; Sample displays strong low angle scattering	5, Col-Pol Crystal 1 hr and 32 minutes after mixing 	r=270 nanometer PMMA Colloid and Polystyrene Polymer; (Mw =3.9e6); Volume fraction Colloid: 0.325 Polymer concentration is Cp =1.055 mg/cm3 Solvent is cis-decalin and tetralin – index match particles
2, Silica Fractal  Soon after constituent combination.	Silica Ludox TM50 Dupont in water, D=22 nanometers, volume fraction = 1.5e-4, Salt is NaCl (4M)	6, Col-Pol Gel  Col-Pol Gel , 1 day 23 hrs and 40 minutes after mix	ASM8 (r=324.7 nanometer) Colloid and Polystyrene Polymer (Mw =1.98e6, Radius = 38 nanometer, Size ratio is 0.12 (polymer to colloid); Volume fraction is Colloid: 0.25; Polymer concentration is Cp =5.0 mg/cm3. Solvent is cis-decalin and tetralin – index match particles. Sample displays a near static scattering ring at low angles.
3, Polystyrene Fractal  Polystyrene Fractal gel: high-mag image 10 days after constituent combination.	Polystyrene (IDC white sulfate 21 nanometer diameter) in water (63% D2O, balance H2O). Volume fraction 8.2e-6; Salt Solution: MgCl2 (0.1 M) in Water (63% D2O, balance H2O)	7, AB_{13} Binary Crystal  AB_{13}: High-mag image.	Vol. Fraction of A (r=415nm) is 0.115, and Vol. Fraction of B (r=237.6nm) is 0.41. Radius ratio is 0.5725. Solvent is cis-decalin and tetralin – index match particles. Crystallization time 5 days
4, AB_6 Binary Crystal  Color image of the AB_6 test cell.	Vol. Fraction of A (r=324.7nm) is 0.4167, and Vol. Fraction of B (r=130nm) is 0.1254. Radius ratio is 0.4004. Solvent is cis-decalin and tetralin –index match particles Crystallization time 2 days.	8, Colloidal Glass  Colloidal Glass sample, 2 hrs 33 min after mix.	0.508 micron poly-methyl methacrylate (PMMA) colloid Total volume fraction is : 0.612 Solvent is cis-decalin and tetralin – index match particles Crystallization time: a few days

Table 1. PCS Flight Samples Details, and some sample pictures.

Four classes of colloidal materials were studied. These include 1) ordered binary crystalline samples; binary colloidal crystal alloy samples are dispersions of two different size particles in an index-matching fluid; 2) mixtures of colloidal particles with other species, primarily polymers, which induce a weak attractive interaction allowing us to precisely tune the phase behavior of the mixtures; Colloid-polymer mixture samples are solutions of mono-disperse particles mixed with a mono-disperse polymer in an index-matching fluid, where the phase behavior—solid, liquid, and gas—is controlled by the concentration of the colloid, the concentration of the polymer, and the relative size of the colloid and the polymer; 3) highly disordered, but very tenuous fractal structures which possess their own unique symmetries and their own unique properties. Fractal gels may form when charged colloids have their electrostatic repulsions screened out by the addition of a salt solution, allowing aggregation. These can be formed at very low volume fractions, and the highly tenuous aggregates formed exhibit a remarkable scaling property: their structure appears the same on all length scales up to a cluster size, and so can be described as a fractal. A polystyrene, and a silica fractal sample were studied; and 4) colloidal glass samples; consider a hard sphere colloid system: as the particles are packed together, if one of them wants to move, its neighbors have to cooperate. As they are packed even tighter, more of the particles have to cooperate for any to move. When all of the particles in the sample have to cooperate, the sample is essentially a solid—this explains what happens with glass, as it is cooled.[7] In the earlier PHaSE flight, it was found that samples which were glasses in 1g crystallized immediately in μg; this sample is to help examine the existence of colloidal glasses in μg.

Thus, there are eight test cells: two binary alloy crystals (one AB_6, one AB_{13}), three Col-Pol systems (one near critical point in two-phase region, one gel-like sample and one crystal sample), two fractal gel systems (one polystyrene, and one silica), and one colloidal glass system.

Initiation of an investigation on each colloid sample occurs when it is homogenized (that is, stirred up via the oscillation of the sample cell within its bearings or mixed with a special fluid combination device) to evenly distribute its suspended particles and then allowed to sit for days, weeks, or months on orbit. During this interval, particles in the samples will organize themselves—that is, self-assemble—into crystal-like or gel-like arrangements. Meanwhile, the laser light scattering and visual imaging diagnostics are utilized to gather structural information about the samples during this crystallization or gelation process. All samples, except the two fractal gels, can be rehomogenized (reinitiated) to repeat a measurement at another sampling rate or to utilize a different measurement technique to examine their behavior in a complementary way. The fractal gels can only be run once. For all samples, measurements were taken every few seconds immediately after homogenization and then with less frequency (typically for a few hours each week) over a several-month period, with on-going experimentation taking place for over several months. The measurements readily provided the information on the growth, the size, and the type of structures formed.

Results

Quantitative data from the dynamic and static light scattering, Bragg scattering, low angle dynamic and static light scattering, and color imaging were obtained; detailed data analysis in each of the research areas (binary colloidal alloys, colloid-polymers, fractal gels, and colloidal glass) is still underway. The latest research updates can be found in the PI website[6] related to this PCS project. The following descriptions illustrate the kind of data obtained from the various samples. Table 1 also shows some color images of these various samples.

Binary Colloidal Crystal Alloy

It is known that under appropriate conditions, monodisperse colloidal particles can self-assemble into crystalline structures with long range periodic order,[8] driven solely by entropy. If particles of different diameters are mixed together, the same entropic effects can lead to the self-assembly of binary alloy crystals.[9,10]

Under certain conditions, it has been found that "hard-sphere" particles (colloidal PMMA) at size ratio 0.58 formed both the AB_2 and the AB_{13} superlattice structures. Several different crystalline structures have been observed, and more are predicted to occur[11].

In PCS, we studied two samples, one an AB_{13} structure, the second an AB_6 structure. In both cases, by monitoring the evolution of the crystallization of each sample, we learned new information that is not accessible in studies performed in 1*g*.

In the case of the AB_{13} sample, we observed unexpected power-law behavior in the growth of the crystals. The origin of these power-law growths is still been investigated as we perform more detailed analysis of the data. The AB_6 sample exhibits quite different growth behavior, and its origin is also still being

investigated. The AB_{13} sample was allowed to anneal for an extended period, and we obtained remarkable static scattering, showing more peaks in the powder pattern than have ever been observed before. This suggests that gravity plays an important role in the ultimate size and morphology of the crystals.

Colloid-Polymer Mixtures

The addition of the polymer induces weak attractive interactions between the colloidal particles by the depletion mechanism, leading to a rich phase behavior for the colloidal particles. Several such systems, including emulsion droplets,[12] charge-stabilized polystyrene spheres,[13] and polymethylmeth-acrylate (PMMA) particles[14] have been studied. As the strength of the attractive interaction is increased by increasing the polymer concentration, the fluid-solid coexistence extends over an increasing range of colloid concentrations.

Colloid-Polymer Crystal:

Initial analysis of the data shows that the colloids phase separate into glassy or disordered regions, followed by crystallization of the glassy regions. We expect to be able to extract useful data about the growth mechanism of the crystals, and to be able to contrast this with the behavior of purely repulsive systems.

Colloid-Polymer Gel:

This sample behaved in an unexpected way; it formed a solid gel, even though we had planned to have a sample that would be in the state that we call the fluid-cluster phase. However, there are also very interesting features of gelled samples that we were able to study with this sample. We studied the solid-like behavior of the gel, and we were able to use the rheology capability of the PCS apparatus to estimate the elastic modulus of the sample. This will be compared to similar samples prepared on the ground, where we can measure the elastic modulus directly. In addition, this gel was found to age, and become slower dynamically, within a short period of time after gelation similar to the aging behavior observed in fractal gels on earth.

Colloid-Polymer Critical Point:

This sample produced a beautiful exhibit of spinodal decomposition over four decades in length scale, from 1 micron to 1 cm. We were able to follow the growth of the length scale with time over the full range of length scales, and the results will be compared to theoretical predictions.

Fractal Gels

The irreversible aggregation of colloidal particles leads to the formation of highly tenuous, but mechanically rigid structures with scale invariant or fractal symmetry.[15] The physical properties of such objects should directly reflect this scale invariance and these experiments will probe these properties. They will focus on the mechanical properties, and thus will measure the dynamics of the fractal objects. If the aggregation is allowed to proceed, unimpeded by the effects of sedimentation, the fractal aggregates will ultimately form a continuous network, or a colloidal gel.[16] This gel will have unique properties, reflecting the fractal scaling of the individual aggregates at shorter length scales, and the mechanical properties of a collection of fractal aggregates at larger length scales. The colloidal polystyrene, and silica gel samples are to study these aggregation characteristics, and test whether or not the aging behavior is unique to polystyrene gels.

The polystyrene fractal sample, which had a very low volume fraction, never gelled in the time it was observed. However, it did produce very interesting results, allowing us to measure the internal vibrational modes of the fractal structure, even though it did not fully gel. This was because the fractal clusters grew so large, which would not be feasible in ground-based experiments. These results show that the theoretical models that describe a gel also work for fractal aggregates, provided they are sufficiently large, and provided the range of scattering angles required can be accessed. In addition, the inhomogeneities visible in the polystyrene sample, quite early in its growth were very puzzling; further ground based experiments based on these results, uncovered the existence of certain limits to the lowest volume fraction at which such gels can be formed in microgravity and in 1g; these ground experiments are in progress toward corroborating the flight experimental results.

The silica sample probably did gel, as expected. This sample was at a higher volume fraction, and so gelled in a shorter time than the polystyrene. This is first time a silica gel has been formed under these low volume fractions, because it is impossible to do this on earth where gravity would crush the gel.

Colloidal Glass:

In an earlier PHaSE flight, it was found that samples which were glasses in 1g crystallized immediately in μg; this sample is to help examine the existence of colloidal glasses in μg. The sample that was flown was a mixture of two glassy samples that had been flown in previous PHaSE flights. This PCS sample

was observed to crystallize rapidly in space; however, preliminary observations suggest that it will crystallize at 1g as well. The origin of this behavior is not clear; it is possible that the sample was poorly mixed, and this left some regions where crystals nucleated more easily, resulting in the crystallization. The colloidal glass flight sample results, and the tests with the colloidal glass sample in 1g (in the engineering unit at Harvard,) indicate that the mixing procedures must be carefully examined for the glass samples. Further experiments were needed on earth after the flight sample was returned. These tests are in progress. These are very critical for upcoming PCS+ experiments.

Need for microgravity, and long duration microgravity:

The formation of colloidal crystals is strongly affected by sedimentation; this is most graphically demonstrated by the results of the experiments of Chaikin and Russel, who showed that the morphology of colloidal crystals grown in space is completely different from that grown on earth.[17]

As the crystals sediment, the shear of the fluid flowing past their edges is sufficient to destroy them. In addition, the sedimentation time of the crystals rapidly begins to compete with the diffusion time of the accreting particles, significantly changing the growth mechanism. While some of this effect can be mitigated by buoyancy matching, this is not completely effective, even at the best level of buoyancy match that can be achieved. By calculating the effective Peclet number, (the ratio of the diffusion time scale of the particles, to the settling time scale of the crystal,) it can be shown that the size of the crystals, $R_{C,\max}$ that can be formed varies inversely as the square root of both g, the gravity, and $\Delta\rho$, the residual density difference after density-matching. That is, if we improve the buoyancy match by two orders of magnitude, the size of the crystals will increase by one order of magnitude; by comparison using the standard non-buoyancy matched fluids, but doing the experiment in microgravity gains an additional 3 decades; this is consistent with what is seen in the CDOT and PHaSE experiments.[17] Combining the buoyancy match and microgravity will produce crystals of remarkable sizes.

These steady streams of PCS data from every sample from the microgravity environment are of great scientific value; incidentally, a visually interesting example is the time-lapse movie of the de-mixing of the colloid-polymer critical point sample. The spinodal decomposition in this sample, in the absence of gravity, over four decades of length scale, (from 1 micron to 1 centimeter,) is being analyzed in detail. Such behaviors cannot be observed in these samples on earth because sedimentation would cause the colloids to fall to the bottom of the cell faster than the de-mixing process could occur. That is, phenomena such as this col-pol spinodal decomposition, and crystallization of samples that remain as glass in 1g, can be studied only in 'microgravity'.

In addition, the 'long durations' over which this microgravity environment is available in the ISS, allows us to zero in on the initial, and final, growth and coarsening behaviors of all the samples; and, e.g., in the case of fractals, the long duration microgravity enabled us to study them at extremely low concentration limits, at which the physical processes of interest are extremely slowed down.

Thus, both, the microgravity and the long duration over which it is available facilitated these studies on the growth and coarsening characteristics of binary crystals, col-pol crystals, gels, fractals, colloidal glasses.

In this context, it is worth pointing out the capabilities of another facility, the LMM (Light Microscopy Module), that is being developed and built by NASA GRC; the LMM will provide the following exciting capabilities: It is a reusable experiment platform that provides confocal- and video-microscopy, laser tweezers, and spectrophotometry, as well as the possibility to study hundreds of samples. For example, we can study 120 or more sample cells, each around 2.5mm dia. and 150 microns thick (~0.75 microliter volume). We can study the structure and dynamics of various systems in real space, allowing us to probe local structure in unprecedented detail. The details and the capabilities of the LMM,[18–21] and upcoming experiments such as PCS-2[22] experiments are described in the references.

PCS Hardware failure and recovery:

On Sunday, February 24, 2002, at the onset of a scheduled operational run, the PCS flight system computer failed to boot up. After various intense recovery attempts, including one on Wednesday, March 20, 2002, in which an ISS crew member, Carl Walz, executed a set of uniquely planned, designed, and verified procedures in which he electrically jumper-connected a monitor and keyboard borrowed from the Human Research Facility (HRF) presently on-board ISS, to the PCS flight system computer to access and modify possible corrupted PCS Basic Input/Output System (BIOS) settings, but this recovery operation was without success. In the week following the March 20th recovery attempt, based on several logistics and risk factors, GRC and the ISS program agreed to terminate

any further on-orbit recovery actions for the PCS flight hardware.

The hardware has been brought down for failure analysis, and it is being fixed and is ready for the next planned activity with the PCS+ experiments by Profs. Paul Chaikin, and William Russel of Princeton University. Despite the various successes described earlier, with each and every sample type investigated, early termination of PCS resulted in some significant missed opportunities:

The measurements of the growth of the AB_6 sample at early times are obscured by scattering from the liquid, and the data we have were not obtained at the optimum time intervals during the growth process; further experiments are needed to investigate this more closely. This would provide critical insight into the nature of the nucleation and growth process of the crystals, one of the key aspects of the formation of these structures. Similarly, it is essential to repeat the measurements of the early stages of the AB_{13} crystals to confirm the unexpected power laws observed.

With the col-pol gel samples, further experiments to extend the range of frequencies over which the elastic modulus was measured are desirable. Rheology measurements as a function of time after the gelation and during the period of aging will provide valuable insights into the mechanisms leading to structural changes. These would provide important additional insight into the nature of the gel. With the col-pol crystal sample, further experiments are desirable to confirm the important observation of the order of the phase separation and crystallization. With the col-pol critical sample, we would like to repeat the test for confirming certain aspects of the set of data we obtained, and also obtain a second "movie" of the spectacular spinodal decomposition behavior in these samples in microgravity.

With the fractal samples, we would like to probe the aging of the silica gel, and compare the results to that of polystyrene gels; this was one of the main motivations of the experiment. The aging of a gel made from a hard material, silica, could be dramatically different from that of a soft sample, polystyrene. This conjecture could not be tested.

Thus, the PCS hardware remains very valuable for these and other such samples in future. While the ISS provides 'long durations' of 'low-gravity' environment, the PCS hardware/facility provides enormous capabilities to study the bulk properties of the colloidal dispersions. This is truly an advanced diagnostic 'facility' that is conducive for a very large class of fluids, condensed matter physics, and biological research work.

Summary

Physics of Colloids in Space (PCS), the first fluids physics experiment that was carried out onboard the ISS has given us tremendous amount of fresh scientific data and knowledge. Four classes of colloids were examined in these experiments, forming binary crystals, col-pol gels and crystals, fractal gels, and glasses. Settling of the particles and aggregates in 1g affects these experiments in various ways; the long durations of microgravity available in the ISS helped unmask these unique data by avoiding such settling, and by extending the experimentation time, thereby allowing us to choose the various samples and study the various growth and coarsening characteristics associated with them.

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13. ABSTRACT ***(Maximum 200 words)***

The Physics of Colloids in Space (PCS) experiment was accommodated within International Space Station (ISS) EXpedite the PRocessing of Experiments to Space Station (EXPRESS) Rack 2 and was remotely operated from early June 2001 until February 2002 from NASA Glenn Research Center's Telescience Support Center (TSC) in Cleveland, Ohio, and from the remote site at Harvard University in Cambridge, Massachusetts. PCS was launched on 4/19/2001 on Space Shuttle STS-100. The experiment was activated on 5/31/2001. The entire experimental setup performed remarkably well, and accomplished 2400 hours of science operations on-orbit. The sophisticated instrumentation in PCS is capable of dynamic and static light scattering from 11 to 169 degrees, Bragg scattering over the range from 10 to 60 degrees, dynamic and static light scattering at low angles from 0.3 to 6.0 degrees, and color imaging. The long duration microgravity environment on the ISS facilitated extended studies on the growth and coarsening characteristics of binary crystals. The de-mixing of the colloid-polymer critical-point sample was also studied as it phase-separated into two phases. Further, aging studies on a col-pol gel, gelation rate studies in extremely low concentration fractal gels over several days, and studies on a glass sample, all provided valuable information. Several exciting and unique aspects of these results are discussed here.

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